

## PATENT SPECIFICATION



Convention Date (Germany) : March 26, 1938.

526,839

Application Date (In United Kingdom) : March 25, 1939.

No. 9420/39.

Complete Specification Accepted : Sept. 26, 1940.

## COMPLETE SPECIFICATION

## Process for the manufacture of Conversion Products of High Molecular Carbohydrates of the Starch groups

We, I. G. FARBEININDUSTRIE AKTIEN-GESELLSCHAFT, a joint stock company organised under the laws of Germany, of Frankfort-on-Main, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

10 This invention relates to a process for the reduction of carbohydrates of the starch group.

Dextrine and similar carbohydrates are produced from starch and like high molecular carbohydrates by the action of dilute acids, diastase, ferments or dry heat. Although pure dextrine is said not to reduce Fehling's solution, the technical dextrines produced according to one of the 20 above methods still have a reducing action which, in many instances, is of disadvantage in their practical application.

In accordance with the present invention non-reducing dextrine preparations 25 can be prepared in a simple and economical manner on a technical scale by catalytic hydrogenation of carbohydrates of the starch group. According to the process of the invention, carbohydrates of the starch 30 group such, for instance, as starch of varying origin, or inulin, or also the still high molecular degradation products thereof such, for instance, as dextrine, amyloses, British gum and so on, are treated in solution or suspension with hydrogen in the presence of a catalyst under elevated pressure and mild conditions of temperature, preferably temperatures between about 100° and about 180° C. The reaction 40 conditions (pressure, temperature, concentration, time of reaction and so on) can be varied within certain limits. In general it is of advantage to have the temperature as low as possible, even if it takes more time for the reaction to be completed.

As liquids in which the starch or like substances are dissolved or dispersed in this process, may be mentioned, for 50 instance, water or methyl alcohol. The catalysts are the usual ones in the art of hydrogenating organic substances; they may be activated, if desired, in known

manner. The hydrogen pressure is preferably higher than 20 atmospheres, the best results so far having been obtained at pressures of 30 to 40 atmospheres. Higher pressures are, however, not excluded.

As above mentioned, the process of the invention can be applied not only to starch, but also to transformation products thereof which are not too low molecular and which have been obtained therefrom, for instance by hydrolysis or by heating. When these products, for example commercial dextrines, are subjected to the 60 present process, their molecular weight is only slightly diminished so that they retain their valuable properties, whilst at the same time their reducing action 70 towards Fehling's solution is destroyed.

The products obtainable according to the process of this invention are of dextrine-like nature. In contradistinction to the commercial dextrines 75 they do not, however, reduce Fehling's solution and are of pure white colour. In consequence thereof, these products and preparations made therefrom are especially suitable in the textile and dyeing industries, for example in the preparation of sizes, finishes and printing pastes 80 and so on.

The products are of special value in the treatment of textiles which have been 85 dyed with vat dyestuffs.

Such dyes are often damaged when treated with alkaline liquids as in washing with soap and soda, in the presence of reducing agents. The shade of the dyeing 90 is changed and the dyestuff bleeds into white parts of the goods (for instance, discharges on printed articles), or into white goods which are simultaneously washed. Such disadvantages do not occur 95 when using the present products which have no reducing action.

The invention is illustrated by the following examples, the parts being by weight:—

## EXAMPLE 1.

100 parts of potato flour and 200 parts of water are stirred at 170° C. in the presence of a nickel catalyst in an autoclave filled with hydrogen at about 30 105 atmospheres for about 15 to 20 hours,

until the aqueous solution can be readily filtered from the catalyst. After evaporation to dryness there is obtained a white dextrinous powder which is soluble in water. It gives a blue reaction with iodine solution, but no longer swells like starch and does not reduce Fehling's solution.

The product may be used, for instance, 10 in a finishing preparation for goods which have been dyed with Hydron blue (compare Schultz Dyestuff Tables, 6th edition, page 257, No. 748). The goods are well filled and of a soft handle. On washing 15 the goods simultaneously with white goods in an aqueous bath containing 5 grams of soap and 3 grams of soda per litre, the shade of the dyeing is not changed, nor can any bleeding into the 20 white goods be observed.

**EXAMPLE 2.**

100 parts of so-called "soluble starch" 25 in 100 parts of water are treated as described in example 1 for 4 hours at 170° C. The resulting product has similar properties to the product of example 1.

**EXAMPLE 3.**

80 100 parts of rice starch or maize flour are hydrogenated in the presence of 200 parts of water for about 2½ hours at 180° C. as described in example 1. The resulting product is very similar to those 35 obtained according to the foregoing examples.

**EXAMPLE 4.**

100 parts of commercial yellow dextrine 40 and 200 parts of water are treated with a nickel catalyst and hydrogen at 20 to 50 atmospheres for 1 to 1½ hours at 140 to 150° C. or for about 4 hours at 130° C. After separating from the catalyst a clear colourless dextrine solution is obtained

which no longer reduces Fehling's 45 solution.

**EXAMPLE 5.**

Technical amylose prepared from potato flour is treated as described in example 3. The white powder obtained in this manner 50 has similar properties to the product obtained according to example 1.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:

1. Process for the manufacture of non-reducing dextrine preparations which comprises treating a carbohydrate of the 60 starch group in suspension or solution catalytically with hydrogen at elevated pressure and temperature.

2. Process for the manufacture of non-reducing dextrine preparations which comprises treating a carbohydrate of the starch group in solution or suspension catalytically with hydrogen at elevated pressure and temperatures of from 100— 70 180° C.

3. Process as claimed in claim 1 or 2 in which starch is employed.

4. Process as claimed in claim 1 or 2 in which dextrine is employed.

5. Process for the manufacture of non-reducing dextrine preparations substantially as described in the examples.

6. Non-reducing dextrine preparations whenever prepared or produced by a process claimed in any of the preceding 80 claims or by any process which is an obvious chemical equivalent thereof.

Dated the 25th day of March, 1939.

CARPMARLS & RANSFORD,  
Agents for Applicants,  
24, Southampton Buildings, London,  
W.C.2.